vinylcarbene toward planar structure.

Conclusion

We applied the concept of the cyclic orbital interaction in acyclic conjugation to complete the relation of the number of electrons with the delocalizability of electrons in acyclic conjugated systems of four p-orbitals (Table I). The $4n + 2$ ($n = 0, 1$) π electrons delocalize in the cross-conjugated systems and localize in the linear-conjugated ones. The $4n$ ($n = 1$) π electrons delocalize in the linear-conjugated systems and localize in the cross-conjugated ones. These conclusions remind us of the Hückel–Möbius concept for cyclic polyenes or annulenes. The cross conjugation can be compared to the Hückel conjugation while the linear conjugation can be compared to the Möbius conjugation. The results of ab initio molecular orbital calculations at the Hartree–Fock levles on the substituted vinylcarbenes and conjugated biscarbenes as the 4e/4p models and others can be understood in terms of phase property of the orbitals involved in cyclic interactions for electron delocalization. Although the Hartree–Fock calculations are thought to reveal the fundamental features of these species in the present comparative study, more sophisticated molecular orbital calculations, especially including electron correlation, are necessary for quantative studies. The clear-cut difference between the cross- and linear-substituted vinylcarbenes or conjugated biscarbenes remains to be experimentally examined in future.

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Electrochemical Generation of Reactive Nitrogen Species. 10.[1] Anodic Amination of Tetrahydrofuran

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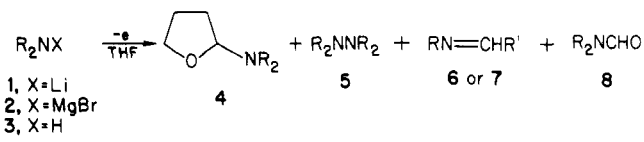
Anodic oxidation in THF of aminyl anions (lithium amides and aminomagnesium bromides) together with free aliphatic amines efficiently afforded 2-aminotetrahydrofurans. A similar reaction took place in the anodic oxidation of *N*-lithiolactams. These reactions are the first examples of anodic amination of an aliphatic saturated ether.

Studies on reactive intermediates seem to be very important to clarify reaction mechanisms and also to design useful synthetic reactions. It is known that various kinds of reactive intermediates are generated by electrochemical reactions.[2] Their reactivities are different from those of nonelectrochemically generated ones in many cases. Although there have been a number of studies on electrochemically generated reactive carbon species, relatively few studies on nitrogen ones have been reported.[3] From these viewpoints, we have reported electrogeneration and reactivities of electron-deficient nitrogen species such as nitrenes,[4] aminonitrenes,[5] nitrenium ions,[6] and aminyl radicals.[7]

In this work, the anodic oxidation of aminyl anions in the form of lithium amides (**1**) and aminomagnesium bromide (**2**) together with free aliphatic amines (**3**), such as alicyclic and open-chain secondary amines, in tetrahydrofuran was studied comparatively. Although a number of electrochemical studies on aliphatic amines have been performed,[8] there has been only one report dealing with anodic oxidation of their aminyl anions.[9] In this case, N,N-coupling products were formed in a moderate yield. In addition, various electrochemical studies on Grignard compounds have been reported,[10] whereas no report has dealt with nitrogen analogues, **2** so far.

Scheme I



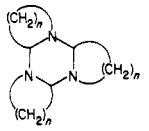
The electrolysis was carried out mainly at a constant current in an undivided cell equipped with two platinum electrodes under a nitrogen atmosphere. Anhydrous tetrahydrofuran containing lithium perchlorate or tetra-*n*-butylammonium tetrafluoroborate was used for electrolytic solution. During electrolysis, the temperature was maintained at ca. 0°C. The results are summarized in Table I.

(1) Part 9: Fuchigami, T.; Nonaka, T. *J. Electrochem. Soc. Jpn.* **1985**, *53*, 582.
(2) Nonaka, T.; Fuchigami, T. *J. Synth. Org. Chem. Jpn.* **1979**, *37*, 372.
(3) Fuchigami, T.; Nonaka, T. *J. Electrochem. Soc. Jpn.* **1984**, *52*, 19.
(4) (a) Fuchigami, T.; Nonaka, T.; Iwata, K. *J. Chem. Soc., Chem. Commun.* **1976**, 951. (b) Fuchigami, T.; Nonaka, T. *Chem. Lett.* **1977**, 1087.
(5) (a) Fuchigami, T.; Sato, T.; Nonaka, T. *J. Electrochem. Soc. Jpn.* **1983**, *51*, 133. (b) Fuchigami, T.; Sato, T.; Nonaka, T.; Lee, J.-K. *J. Chem. Soc. Jpn.* **1984**, 1769. (c) Fuchigami, T.; Sato, T.; Nonaka, T. *Electrochim. Acta* **1985**, *30*, No. 12.
(6) Fuchigami, T.; Toki, T.; Iwaoka, T.; Nonaka, T. *J. Electrochem. Soc. Jpn.* **1979**, *47*, 627.
(7) Fuchigami, T.; Iwaoka, T.; Nonaka, T.; Sekine, T. *Chem. Lett.* 1978, 1437; *Bull. Chem. Soc. Jpn.* **1980**, *53*, 2040.
(8) (a) Mann, C. K.; Barnes, K. K. "Electrochemical Reactions in Nonaqueous Systems"; Marcel Dekker: New York, 1970); p 269. (b) Shono, T. "Electroorganic Chemistry as a New Tool in Organic Synthesis"; Springer-Verlag: Berlin, Heidelberg, 1984.
(9) Bauer, R.; Wendt, H. *Angew. Chem.* **1978**, *90*, 214.
(10) (a) Evans, W. V.; Lee, F. H. *J. Am. Chem. Soc.* **1934**, *56*, 654. (b) Gillet, I. E. *J. Electrochem. Soc.* **1961**, *108*, 71.

0022-3263/86/1951-0366$01.50/0

Table I. Anodic Oxidation of Lithium Amide, Aminomagnesium Bromide, and Free Amine (1-3) in Tetrahydrofuran

run	R_2NX (1-3) $R_2N\sim$	X	(mmol)	no.	anolyte[a]	current density (A/dm^2)	charge passed (F/mol)	convd, of 1-3, %	yield,[b] % 4	5	6[c] or 7[d]	8[e]
1		Li	(5)	**1a**	A	0.21	1.0	23	72	3	6	0
2		MgBr	(20)	**2a**	*f*	0.84	1.0	15	2	22	15	0
3		H	(20)	**3a**	B	0.84	1.0	27	81	2	*g*	0.3
4		H	(20)	**3a**	A′	0.84	1.0	21	61	2	4	0
5		H	(20)	**3a**	A′	0.84	4.0	70	50	1	0	0.1
6		Li	(5)	**1b**	A	0.21	1.0	16	16	trace	trace	0
7		MgBr	(20)	**2b**	*f*	0.84	1.0	57	0	8	1	0
8		H	(20)	**3b**	B	0.84	1.0	68	21	0	trace	0
9		Li	(5)	**1c**	A	0.21	1.0	35	22	0	0	0.2
10		Li	(5)	**1c**	A	0.21	2.0	52	42	0	trace	0
11		MgBr	(20)	**2c**	*f*	0.84	1.0	75	trace	0.3	*g*	0
12		H	(20)	**3c**	A′	0.84	1.0	79	1	0	5	0
13	n-$Bu_2N\sim$	Li	(5)	**1d**	A	0.21	1.0	15	30	trace	*g*	0
14		MgBr	(20)	**2d**	*f*	0.84	1.0	23	0	7	*g*	0
15		H	(20)	**3d**	A′	0.84	1.0	56	3	0	*g*	3

[a] A: 0.25 M $LiClO_4$ (20 mL), A′: 0.8 M $LiClO_4$ (20 mL), B: 1.0 M n-Bu_4NBF_4 (20 mL). [b] Based on consumed 1-3. [c] 6:  [d] 7:  (n = 3, 4) [e] Based on starting 1-3. [f] No supporting electrolyte. [g] Not determined.

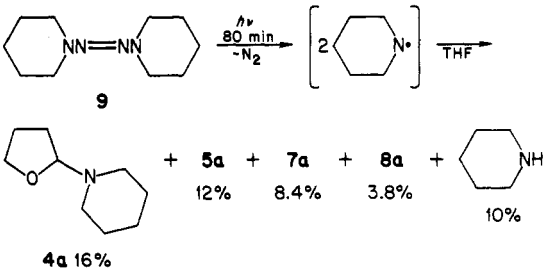
Surprisingly, 2-aminotetrahydrofurans **4** were formed as main products while N,N-coupling products, hydrazines **5**, were rarely obtained (Scheme I). It is noticeable that the anodic amination of tetrahydrofuran took place efficiently from **1** and **3**, while such amination from **2** occurred only to a small extent. In the latter case, the N,N-coupling took place favorably (runs 2, 7, 14). In the electrolysis of pyrrolidine, **4** was obtained in 42% yield from **1**, while **4** was scarcely formed from **3** (runs 10, 12). These results indicate that reactivities of anodically generated reactive nitrogen species greatly depend not only on the molecular structures of the parent amines but also on their generation sources (**1**-**3**).

Since the yield of 2-piperidinotetrahydrofuran based on the consumed piperidine was very high (72% and 81% from **1** and **3**, respectively (runs **1**, **3**)), the anodic amination seems to occur exclusively in these electrolytic reactions.

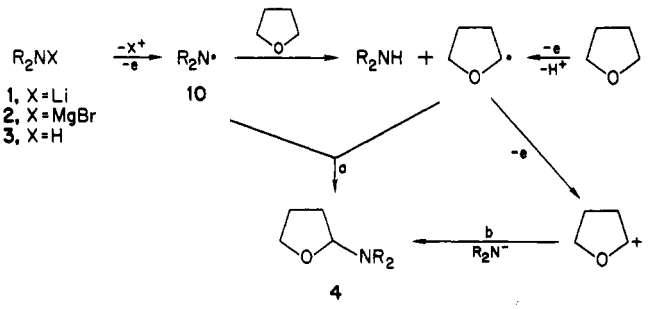
Wendt et al.[9] reported that anodic oxidation of lithium amides was carried out in tetrahydrofuran at a constant potential by using a divided cell to give N,N-coupling products exclusively. Although our electrolytic conditions are a little different, our results are quite different from theirs. Shono and Matsumura reported anodic methoxylation of tetrahydrofuran.[11] However, the yield was relatively low. Our results appear to be somewhat similar to their reaction.

Since an imine (**6**) and/or imine trimers (**7**) were detected as byproducts from the anolyte, the reaction intermediates generated in the course of the electrode process seem to be aminyl radicals. In order to clarify the mechanism of this novel anodic amination, constant potential electrolysis of **1** and photolysis of a 2-tetrazene were investigated by using piperidine as a model compound. Lithium piperidide was electrolyzed at a potential (+0.6 V vs. Ag/0.1 M Ag^+, 0.55 F/mol), where tetrahydrofuran could not be oxidized. In this electrolysis, the amination

Scheme II



Scheme III

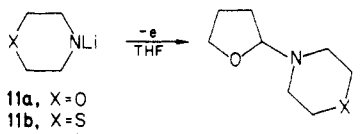


occurred mainly and **4a** (23% yield based on the consumed the amine) together with small amounts of **5a**, **7a** (5%), and **8a** (16%) were formed. Photolysis of 2-tetrazene **9** in tetrahydrofuran with a high pressure mercury lamp also provided **4a** as the main product as shown in Scheme II. Such photochemical amination of tetrahydrofuran has not been observed so far.[12] In both the anodic and photochemical reactions, formamide derivatives **8** were found to be formed as byproducts other than **6** and **7**. However, at the present time, the formation mechanism of **8** has not

(11) Shono, T.; Matsumura, Y. *J. Am. Chem. Soc.* **1969**, *91*, 2803.

(12) Most recently, Newcomb and Williams have investigated the photochemical decomposition of N^1,N^4-dicyclobutyl-N^1,N^4-dimethyltetrazene in THF; however they have not detected any aminated THF: Newcomb, M.; Williams, W. G. *Tetrahedron Lett.* **1984**, *25*, 2723.

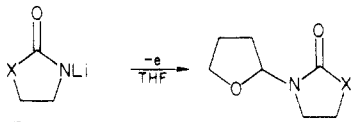
Scheme IV



11a, X=O
11b, X=S
11c, X=NEt

12

Scheme V



13a, X=CH_2
13b, X=O

14

Table II. Anodic Amination of Tetrahydrofuran[a]

run	lithium amide	current density (A/dm^2)	charge passed (F/mol)	convn of 11 or 13, %	yield,[b] %
16	**11a**	0.21	1.0	50	36
17	**11b**	0.40	1.0	33	24
18	**11c**	0.30	1.0	46	9
19	**13a**	0.32	1.0	89	5
20	**13b**	0.17	0.5	53	26

[a] Electrolyte: 20 mL of 0.25 M $LiClO_4$/THF containing 5 mmol of 11 or 13. [b] Based on the consumed starting amine or lactam.

been clarified.[13] Since the photolysis of tetrazenes is known to generate aminyl radicals , the most plausible reaction intermediates in this novel amination should be aminyl radicals. A tentative mechanism of the anodic amination of tetrahydrofuran is shown in Scheme III. 2-Aminotetrahydrofurans **4** may be formed by cross-coupling of aminyl radicals (**10**) with 2-tetrahydrofuryl radicals [route a]. In addition to route a, this amination also appears to result from another route (b) involving tetrahydrofuryl cations, which is a similar mechanism proposed for the anodic methoylation of tetrahydrofuran,[11] even though the oxidation potential of tetrahydrofuran is much higher than that of **1–3**, because the anodic potential was not controlled in most of our experiments.[14]

To our knowledge, this reaction is the first example of anodic amination of an aliphatic saturated ether.

Next, we attempted some extentions of this novel anodic amination to more complicated cyclic amines as shown in Scheme IV. It is known that anodic cleavage of a carbon–carbon bond of *vic*-diamines,[16] 1,2-amino alcohols,[17] and *vic*-diols[18] very often takes place.[19] However, the anodic amination was found to proceed in the case of the lithium amides **11** of these cyclic amines without any cleavage of the carbon–carbon bond to give **12** as shown in Table II (runs 16–18).

Finally, we extended this further using *N*-lithiolactams (**13**) such as pyrrolidone and 2-oxazolidone (Scheme V, Table II; runs 19, 20). Both lithium amides **13** provided tetrahydrofurans substituted by the lactams used (**14**) although their yields were low. It was thus confirmed that anodic amidation of tetrahydrofuran also proceeded in a similar manner.

Experimental Section

^1H NMR spectra were recorded at 60 MHz on a Varian EM 360 NMR spectrometer using $CDCl_3$ and Me_4Si as solvent and internal standard, respectively. IR spectra were obtained with a Hitachi 295 infrared spectrometer. Mass spectra were obtained with a JEOL JMS-D100 GC–mass spectrometer. High-resolution mass spectra were obtained with JEOL JMS-01SC mass spectrometer.

Electrolytic Procedure. An undivided cylindrical cell equipped with a magnetic stirrer and a serum cap for introduction of nitrogen gas was used. Electrolysis was carried out at a constant current using a platinum plate (3 × 3 cm or 4 × 4 cm) as an anode under a nitrogen atmosphere. Electrolytic conditions in each electrolysis are shown in Tables I and II.

(a) Anodic Oxidation of Lithium Amide (1, 11, and 13). To a stirred solution of 5 mmol of amine or amide in 20 mL of 0.25 M $LiClO_4$/anhydrous THF solution was added dropwise 3.2 mL of a 15% *n*-butyllithium–hexane solution below −10 °C. After 30 min of stirring, the temperature was raised to 0 °C and then an electrolytic current was supplied. After the electrolysis, the electrolyte was mixed with 10 mL of water to decompose deposited lithium metal on the cathode. The solution was acidified with hydrochloric acid and was extracted with ether to remove mainly THF. The aqueous layer was made strongly alkaline with sodium hydroxide and then was saturated with sodium chloride. The alkaline solution was extracted 7 times with 30-mL portions of ether and was dried over anhydrous sodium sulfate. The extracts were concentrated by distillation using a Widmer fractionating column and the remaining solution was subjected to GLC analysis.

(b) Anodic Oxidation of Aminomagnesium Bromide (2). To a stirred solution of 10 mL of 2.5 M ethylmagnesium bromide/THF solution was added dropwise 20 mmol of amine at ca. 20 °C. During the addition, the ethane gas evolved almost quantitatively. After the gas evolution had ceased, tetrahydrofuran was added to adjust the whole volume of the electrolyte to 20 mL and a current was suppled. After the electrolysis, the electrolyte was poured into 40 mL of water and the solution was acidified with hydrochloric acid. The solution was extracted with ether to remove mainly THF. The aqueous solution was made strongly alkaline with sodium hydroxide. The resulting suspension was extracted repeatedly with ether and was analyzed in a manner similar to the case of a.

Anodic oxidation of free amines (**3**) were carried out in a similar manner.

Analysis and Identification of the Products. 2-Piperidinotetrahydrofuran (**4a**), 2-pyrrolidinotetrahydrofuran (**4c**), and *N*-formylpiperidine (**8a**) were isolated by preparative GLC and identified by studies of various spectral data. All products including **4a**, **4c**, and **8a** were identified by comparison of the mass fragmentations[20] and retention times with those of authentic samples synthesized separately (see the following section).

GLC analysis was carried out with a stainless steel column (2.25 m × 4 mm) packed with 3% PEG 20M-15% KOH or PEG 20M on 60–80-mesh Celite 545.

Preparation of Authentic Samples. 2-Aminotetrahydrofurans 4 and 12. According to the method similar to that described by Eliel and Daignault,[21] **4** and **12** were prepared from 2-chlorotetrahydrofuran and amines as follows: Hydrogen chloride gas was bubbled into a solution of 25.00 g (0.358 mol) of 2,3-

(13) The formamides **8** were assumed to be formed by the further oxidation of **4**. However, **4** was oxidized under the same conditions as the electrolysis of **1** to give no **8**. Ogawa et al. also observed the formation of formamide derivatives by the oxidation of cyclic secondary amines with peroxodisulfide: Ogawa, K.; Nomura, Y.; Takeuchi, Y.; Tomoda, S. *J. Chem. Soc., Perkin Trans 1* **1982**, 3031.

(14) Anodic oxidation of tetrahydrofuran probably took place in the constant current electrolysis since polymeric products of tetrahydrofuran were obtained in addition to the basic products **4–8**. Anodic oxidation of tetrahydrofuran is known to give polymers of tetrahydrofuran.[15]

(15) (a) Makahama, S.; Hino, S.; Yamazaki, N. *Polymer J.* **1971**, *2*, 56. (b) Dey, A. N.; Rudd, E. J. *J. Electrochem. Soc.* **1974**, *121*, 1294.

(16) Smith, J. R. L.; Masheder, D. *J. Chem. Soc., Perkin Trans 2* **1977**, 1732.

(17) (a) Masui, M.; Sayo, H. *J. Chem. Soc. B* **1971**, 1593. (b) Portis, L. C.; Klug, J. T.; Mann, C. K. *J. Org. Chem.* **1974**, *39*, 3488. (c) Shono, T.; Hamaguchi, H.; Matsumura, Y.; Yoshida, K. *Tetrahedron Lett.* **1977**, 3625. (d) Masui, M.; Ozaki, S. *Chem. Pharm. Bull.* **1978**, *26*, 2153. (e) Masui, M.; Kamada, Y.; Ozaki, S. *Chem. Pharm. Bull.* **1980**, *28*, 1619. (f) Masui, M.; Kamada, Y.; Sasaki, E.; Ozaki, S. *Chem. Pharm. Bull.* **1982**, *30*, 1234.

(18) Shono, T.; Matsumura, Y.; Hashimoto, T.; Hibino, K.; Hamaguchi, H.; Aoki, T. *J. Am. Chem. Soc.* **1975**, *97*, 2546.

(19) Fuchigami, T.; Nonaka, T.; Watanabe, C.; Yoshiyama, A.; Sekine, T. *J. Electrochem. Soc. Jpn.* **1983**, *51*, 812.

(20) The mass spectra were measured with a gas chromatograph–mass spectrometer.

(21) Eliel, E. L.; Daigault, R. A. *J. Org. Chem.* **1965**, *30*, 2450.

dihydrofuran in 80 mL of anhydrous ether at 0 °C until no more was absorbed. Concentration and distillation under reduced pressure provided 26.10 g (68%) of 2-chlorotetrahydrofuran,[22] bp 36–37 °C (16–17 torr); MS, *m/e* 71 (M^+ – HCl).

To a stirred suspension of 0.25 mol of amine and 0.2 mol of anhydrous potassium carbonate in 100 mL of anhydrous ether was added dropwise a solution of 0.1 mol of 2-chlorotetrahydrofuran in 100 mL of ether. After the addition was complete, the mixture was refluxed for 2 h. After cooling, 100 mL of water was added to dissolve the salts. The ether layer was separated and the aqueous layer was extracted 2 times with 100-mL portions of ether. The combined etheral extracts were dried over potassium carbonate. The extracts were concentrated and vacuum distillation of the residue provided pure 2-aminotetrahydrofurans **4** and **12**.

2-Piperidinotetrahydrofuran (4a): yield 57%; bp 65–68 °C (6 torr); ^{1}H NMR δ 1.1–1.7 (m, 6 H), 1.7–2.1 (m, 4 H), 2.1–3.1 (m, 4 H), 3.4–4.1 (m, 2 H), 4.60 (t, 1 H); MS, *m/e* (relative intensity) 155 (M^+, 46), 154 (M^+ – H, 46), 125 (65), 112 (35), 110 (100), 84 (45), 71 (32); calcd for $C_9H_{17}NO$ *m/e* 155.1310, found 155.1313.

2-[1-(2-Methyl)piperidino]tetrahydrofuran (4b): yield 40%; bp 115 °C (27 torr); ^{1}H NMR δ 1.20 (d, 3 H), 1.3–1.7 (m, 6 H), 1.7–2.1 (m, 4 H), 2.2–3.1 (m, 3 H), 3.5–4.2 (m, 2 H), 5.20 (t, 1 H); MS, *m/e* (relative intensity) 169 (M^+, 25), 154 (M^+ – Me, 41), 124 (31), 84 (100), 71 (29); calcd for $C_{10}H_{19}NO$ *m/e* 169.1466, found 169.1461.

2-Pyrrolidinotetrahydrofuran (4c): yield 60% bp 90–93 °C (34 torr); ^{1}H NMR δ 1.5–2.1 (m, 8 H), 2.5–3.0 (m, 4 H), 3.7–4.0 (m, 2 H), 4.70 (t, 1 H); MS, *m/e* (relative intensity) 141 (M^+, 41), 140 (M^+ – H, 39), 111 (43), 96 (100), 71 (64), 70 (41), 43 (43); calcd for $C_8H_{15}NO$ *m/e* 141.1153, found 141.1154.

2-(Dibutylamino)tetrahydrofuran (4d): yield 45%; bp 112–115 °C (12 torr); ^{1}H NMR 0.7–1.1 (m, 6 H), 1.1–1.7 (m, 8 H), 1.7–2.1 (m, 4 H), 2.4–2.9 (m, 4 H), 3.7–4.1 (m, 2 H), 4.90 (t, 1 H); MS, *m/e* (relative intensity) 199 (M^+, 28), 156 (M^+ – Pr, 44), 140 (75), 86 (97), 84 (28), 71 (100); calcd for $C_{12}H_{25}NO$ *m/e* 199.1914, found 199.1936.

2-Morpholinotetrahydrofuran (12a): yield 49%; bp 76–77 °C (4 torr); ^{1}H NMR δ 1.6–2.1 (m, 4 H), 2.3–2.9 (m, 4 H), 3.4–3.9 (m, 6 H), 4.50 (t, 1 H); MS, *m/e* (relative intensity) 157 (M^+, 58), 156 (M^+ – H, 46), 127 (54), 112 (42), 86 (40), 73 (48), 71 (100); calcd for $C_8H_{15}NO_2$ *m/e* 157.1101, found 157.1076.

2-Thiomorpholinotetrahydrofuran (12b): yield 19%; mp 105–107 °C (3 torr); ^{1}H NMR δ 1.7–2.1 (m, 4 H), 2.5–2.9 (m, 4 H), 2.9–3.3 (m, 4 H), 3.7–4.0 (m, 2 H), 4.57 (t, 1 H); MS, *m/e* (relative intensity) 173 (M^+, 20), 114 (20), 90 (20), 73 (50), 71 (38), 45 (100); calcd for $C_8H_{15}NOS$ *m/e* 173.0872, found 173.0867.

2-(*N*-Ethylpiperazino)tetrahydrofuran (12c): yield 18%; bp 91 °C (4 torr); ^{1}H NMR δ 1.07 (t, 3 H), 1.66–2.07 (m, 4 H), 2.2–3.0 (m, 10 H), 3.63–3.95 (m, 2 H), 4.53 (t, 1 H); MS, *m/e* 184 (M^+, 51), 114 (63), 100 (71), 84 (100), 73 (43), 71 (53), 70 (45); calcd for $C_{10}H_{20}N_2O$ *m/e* 184.1573, found 184.1510.

1-(2-Tetrahydrofuryl)-2-pyrrolidone (14a). To a stirred solution of 3.73 g (35 mmol) of 2-pyrrolidone in 30 mL of THF was added 24 mL of 15% *n*-BuLi/hexane solution below –10 °C. After 30 min of stirring, a solution of 3.73 g (35 mmol) of 2-chlorotetrahydrofuran in 5 mL of THF was added to the solution and the mixture was stirred for 1.5 h at 0 °C. After stirring overnight, 10 mL of water was added, the organic layer was separated, and then the aqueous layer was extracted twice with 30-mL portions of ether. The combined extracts were dried over magnesium sulfate and concentrated under reduced pressure. Distillation of the remaining oil provided 2.13 g (39%) of pure **14a** as a colorless oil, bp 105 °C (2 torr): ^{1}H NMR δ 1.66–2.50 (m, 8 H), 3.16–4.00 (m, 4 H), 5.80 (t, 1 H); IR 2950, 2880 (ν_{CH}), 1680 cm^{-1} ($\nu_{C=O}$); MS, *m/e* 155 (M^+, 42), 127 (M^+ – CO, 52), 112 (45), 111 (45), 85 (95), 71 (100). Anal. Calcd for $C_8H_{13}NO_2$: C, 61.91; H, 8.44; N, 9.03. Found: C, 61.78; H, 8.69; N, 9.10.

3-(2-Tetrahydrofuryl)-2-oxazolidone (14b). In a manner similar to the preparation of **14a**, **14b** was synthesized, yield 35%, as a colorless oil: ^{1}H NMR δ 1.66–2.30 (m, 4 H), 2.50 (t, 2 H), 2.66–4.00 (m, 2 H), 4.33 (t, 2 H), 5.53 (t, 1 H); IR 2980, 2930, 2890 (ν_{CH}), 1760 cm^{-1} ($\nu_{C=O}$); MS, *m/e* 157 (M^+, 10), 129 (M^+ – CO, 5), 114 (40), 113 (M^+ – CO_2, 30), 71 (100). Anal. Calcd for $C_7H_{11}NO_3$: C, 53.50; H, 7.05; N, 8.91. Found: C, 53.58; H, 7.15; N, 9.06.

Hydrazine Derivatives 5. 1,1′-Bipiperidines (**5a** and **5b**) and 1,1′-bipyrrolidine (**5c**) were prepared by the oxidation of piperidines and pyrrolidine with sodium peroxodisulfate according to the method reported by Ogawa et al.[13]

An Imine (6) and Imine Trimers (7). 2,3,4,5-Tetrahydropyridine trimer (**7a**),[23] 1-pyrroline trimer (**7c**),[13] and 2,3,4,5-tetrahydro-6-methylpyridine (**6**)[24] were prepared by the reaction of the corresponding amines with *N*-chlorosuccinimide or sodium peroxodisulfate.

Photolysis of 1,2-Dipiperidyltetrazene (9). A 5-mmol sample of tetrazene **9** was irradiated for 80 min in 20 mL of anhydrous THF with a 100-W high pressure mercury lamp below 30 °C under a nitrogen atmosphere. After irradiation, the reaction mixture was subjected to GLC analysis.

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Registry No. **1a**, 4442-11-9; **1b**, 84602-06-2; **1c**, 4439-90-1; **1d**, 25440-26-0; **2a**, 24699-38-5; **2b**, 99705-98-3; **2d**, 99706-01-1; **3a**, 110-89-4; **3b**, 109-05-7; **3c**, 123-75-1; **3d**, 111-92-2; **4a**, 99705-97-2; **4b**, 99705-99-4; **4c**, 99706-00-0; **4d**, 99706-02-2; **5a**, 6130-94-5; **5b**, 66614-22-0; **5d**, 60678-70-8; **6**, 1462-92-6; **7a**, 522-33-8; **7c**, 5981-17-9; **8a**, 2591-86-8; **8d**, 761-65-9; **9**, 2081-14-3; **11a**, 37828-58-3; **11a** (amine), 110-91-8; **11b**, 99706-03-3; **11b** (amine), 123-90-0; **11c**, 99706-04-4; **11c** (amine), 5308-25-8; **12** (X = O), 20024-89-9; **12** (X = S), 99706-06-6; **12** (X = NEt), 99706-07-7; **13a**, 17726-07-7; **13a** (amide), 616-45-5; **13b**, 99706-05-5; **13b** (amide), 497-25-6; **14** (X = CH_2), 73486-65-4; **14** (X = O), 99706-08-8; 2,3-dihydrofuran, 1191-99-7; 2-chlorotetrahydrofuran, 13369-70-5; tetrahydrofuran, 109-99-9.

(22) This compound decomposes very easily even if it is stored in a refrigerater over anhydrous potassium carbonate. This is quite different from 2-chlorotetrahydrofuran.[21]

(23) Schöpf, C.; Komzak, A.; Brawn, F.; Jacob, E. *Liebigs Ann. Chem.* **1948**, *559*, 1.

(24) Archer, J. F.; Boyd, D. R.; Jackson, W. R. *J. Chem. Soc.* **1971**, 2560.